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AN IMPROVEMENT IN THE METHOD FOR DISSOLVING CELLULOSE IN CUPRAMMONIUM SOLUTION FOR FLUIDITY MEASUREMENTS

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ABSTRACT

The use of mixing vials in place of the usual calibrated viscometers as vessels for dissolving cellulose in cuprammonium solution is described. The solution of cellulose is prepared in the vial and poured into a calibrated viscometer for flow measurements. The data given show that the exposure of the solution to the air for the short time required for pouring has no measurable effect on the fluidity.

CONTENTS

I. INTRODUCTION

In determinations of the fluidity of cuprammonium solutions of cellulose, the solution is usually prepared [1] [2] in the viscometer. A steel plunger is placed in the mixture of cellulose and cuprammonium solution and the viscometer rotated end over end. During this operation it occasionally happens that when celluloses, which swell and mix slowly with the liquid, are to be dissolved, the plunger drags cellulose to the ends of the viscometer and plugs the capillary. Also, the notched end of the plunger repeatedly strikes the narrowed end of the viscometer and may break it.

Cuprammonium solutions when in contact with air may be changed because of loss of ammonia and absorption of oxygen [3]. It was largely because of this instability that the solution of cellulose was prepared in the viscometer. It has been found, however, that the solutions of cellulose can be prepared in another vessel and transferred to the viscometer without measurable change in fluidity.

This paper describes an improved technique for dissolving cellulose, in the absence of air or any gas, in mixing vials. The technique permits the reservation of viscometers as instruments for flow measurements only. It is the result of experiments to prepare solutions of cellulose in dimethyldibenzylammonium hydroxide, and as the

[1] ASTM Designation D-539-39T. ASTM Standards, pt. 3, p. 981 (1939).
[2] J. Research NBS **22**, 271 (1939) RP1179.
[3] Ind. Eng. Chem., Anal. Ed. **13**, 322 (May 15, 1941).

xanthate, for fluidity measurements.[4] Data on fluidity measurements obtained with the usual method for effecting dispersion of cellulose in cuprammonium and with this method are presented.

II. PREPARATION OF THE CELLULOSE SOLUTION

Equipment suitable for dispersing celluloses in cuprammonium solutions is illustrated in figure 1. It consists of a mixing vial, *V*, fitted with a glass or iron stopper, *S*, and a glass plunger, *P*. It is desirable that the vial and plunger be so shaped that swollen cellulose will not adhere long to any part of the vial as it is rotated to effect solution.

To prepare a solution of cellulose for fluidity measurements, the vial containing the plunger is half filled with cuprammonium solution, and the required amount of cellulose is added and wetted with the

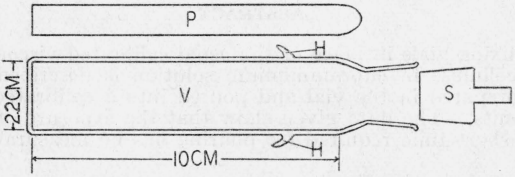

FIGURE 1.—*Mixing vial for effecting dispersion of cellulose in cuprammonium solution.*

V, Glass body of vial; *S*, glass or metal stopper; *P*, glass plunger. Flat end of plunger placed at bottom of vial when filling with cellulose and cuprammonium solution; *H*, hooks for rubber bands used to hold on stopper.

solution. More solution is then added until insertion of the stopper displaces all the air, with some of the cuprammonium solution overflowing. The stopper is held in place with rubber bands passing around the hooks, *H*, and the vial is attached to a wheel and rotated about its long axis at 50 to 55 rpm until solution is completed. It usually is convenient to rotate the vials overnight. While effecting solution, it is well to keep the temperature from rising above 25° C. This can be done by placing the equipment in a room held below this temperature or by mounting the wheel in a trough through which cool tap water is flowing. Measurements made on celluloses dissolved at 13° and 25° C showed that temperature changes within this range during the preparation of the solution had no appreciable effect on the fluidity. After solution is complete, the vial and contents are placed in a constant-temperature bath, and after reaching temperature equilibrium, the contents are poured into a calibrated viscometer at the same temperature and the flow measurements made in the usual manner. While transferring the liquid from the vial to the viscometer, the capillary end of the viscometer should be closed. This may be done by slipping a short piece of plugged rubber tubing over the capillary. The tubing is removed, and the usual observations are made on the moving meniscus as the liquid drains from the viscometer.

[4] J. Research NBS 27, 543 (1941) RP1441.

III. FLUIDITY MEASUREMENTS

The fluidity of the cellulose solution is not changed appreciably in the short time it is exposed to the air by the new technique. This is shown in table 1, which gives the results of measurements made at 21° C on 0.5-percent solutions of celluloses of different viscosity characteristics. The results in the left columns for the three samples were obtained by effecting solution in the viscometers with the metal plungers. The measurements were made with the viscometers used for preparing the solution. The second column gives the results of measurements on solutions prepared in the mixing vials and then transferred to a viscometer for measuring. The reproducibility of the results obtained by the two methods is approximately the same except for sample 3, for which it is somewhat better for solutions prepared in the mixing vials. More consistent results may be expected when using the vials, because of the more thorough mixing.

TABLE 1.—*Fluidity, in rhes, of celluloses of different flow characteristics*

Sample number—					
1		2		3	
Viscometers [a]	Vials [b]	Viscometers [a]	Vials [b]	Viscometers [a]	Vials [b]
2.67	2.72	12.0	12.0	17.9	17.4
2.53	2.74	12.0	12.0	17.5	17.5
2.66	2.71	11.9	12.0	18.0	17.3
2.69	2.62	11.9	11.9	17.3	17.4
2.61	2.61	11.7	11.9	17.6	17.4
2.65	2.68	11.9	12.0	17.7	17.5
2.68	2.61	11.9	12.6	17.4	17.5
2.68	----------	----------	12.3	17.4	17.4
Mean 2.65	2.67	11.9	12.1	17.6	17.4

[a] Cellulose solutions prepared in viscometers.
[b] Cellulose solutions prepared in mixing vials and transferred to viscometers for measurement.

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